References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
DODGE, R. P., TEMPLETON, D. H. & ZALKIN, A. (1961). *J. Chem. Phys.* **35**, 55–67.
HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
HON. P. K., BELFORD, R. L. & PFLUGER, C. E. (1965). *J. Chem. Phys.* **43**, 1323–1333, 3111–3115.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
MUKHERJEE, A. K. & RAY, P. (1955). *J. Indian Chem. Soc.* **32**, 505–509.
STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
ZERNER, M. & GOUTERMAN, M. (1966). *Inorg. Chem.* **5**, 1699–1706.

Acta Cryst. (1985). C**41**, 1728–1730

Structure of (Benzoato-*O,O′*)bis(2-dimethylaminoethanol)copper(II) Benzoate

BY URHO TURPEINEN AND REIJO HÄMÄLÄINEN

Department of Inorganic Chemistry, University of Helsinki, SF-00100 Helsinki 10, Finland

AND MARKKU AHLGRÉN

Department of Chemistry, University of Joensuu, SF-80100, Joensuu 10, Finland

(*Received 7 March 1985; accepted 15 August 1985*)

Abstract. $[Cu(C_7H_5O_2)(C_4H_{11}NO)_2][C_7H_5O_2]$, $M_r = 484{\cdot}05$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}166\,(3)$, $b = 13{\cdot}293\,(7)$, $c = 19{\cdot}274\,(7)$ Å, $V = 2348\,(2)$ Å^3, $D_x = 1{\cdot}37$ g cm^{-3}, $Z = 4$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 10{\cdot}1$ cm^{-1}, $F(000) = 1020$, $T = 293$ K, $R = 0{\cdot}049$ for 1610 observed reflections. The Cu atom has an unusual distorted octahedral environment with two approximately collinear short Cu—N bonds of 2·060 (6) and 2·058 (6) Å, three intermediate Cu—O bonds of 2·126 (6), 2·168 (6) and 2·179 (6) Å, and one longer Cu—O bond of 2·370 (6) Å from the asymmetrically chelated carboxylate group.

Introduction. Several tetrameric complexes have been obtained from reactions between 2-dimethylaminoethanol and Cu^{II} carboxylates (Turpeinen, Hämäläinen & Ahlgrén, 1980; Ahlgrén, Turpeinen & Hämäläinen, 1982). Blue and green crystals crystallized simultaneously during slow evaporation of ethanol solution containing Cu^{II} benzoate and 2-dimethylaminoethanol. The present study shows that the blue form of the complex is the title compound.

Experimental. Blue, rhombic crystal 0·3 × 0·3 × 0·4 mm. Nicolet *P*3 diffractometer, graphite-monochromatized Mo *Kα* radiation. Unit-cell dimensions obtained from 20 reflections in θ range 6–13°. Intensity data $4° < 2\theta < 50°$. ω-scan technique. Scanning speed varied according to the prescanned peak intensities from 2 to 30° min^{-1}. Three standard reflections every 80 reflections showed no significant change in intensity. Lp correction, no absorption or extinction corrections. 2425 unique reflections measured, 1610 with $I > 3\sigma(I)$; index range h 0–10, k 0–15, l 0–22. Structure solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Least squares (*XRAY76*, Stewart, 1976), minimizing $\sum w(\Delta F)^2$, with anisotropic thermal parameters for non-H atoms, isotropic for H atoms of the hydroxyl groups located from difference synthesis. The other 30 H atoms with $U = 0{\cdot}08$ Å^2 included at idealized positions (C—H 1·0 Å) and held fixed. Final $R = 0{\cdot}049$, $wR = 0{\cdot}032$ for observed data (the rejected enantiomeric structure had $R = 0{\cdot}056$, $wR = 0{\cdot}040$); $w = 1/\sigma^2(F_o)$ based on counting statistics. Residual electron density within ±0·3 e Å^{-3}. Largest $\Delta/\sigma = 0{\cdot}56$. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974).

Discussion. The atomic coordinates and temperature factors are listed in Table 1 and interatomic distances

0108-2701/85/121728-03$01.50

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Cu	5535 (1)	5761 (1)	5775 (1)	0·042
O(1)	3431 (6)	5191 (4)	6125 (3)	0·059
O(2)	5160 (6)	7233 (4)	6225 (3)	0·056
O(3)	6678 (5)	4508 (5)	5357 (3)	0·096
O(4)	7648 (7)	5931 (4)	5076 (3)	0·113
O(5)	2591 (5)	7574 (3)	6717 (2)	0·058
O(6)	2344 (5)	6056 (3)	7199 (2)	0·061
N(1)	4323 (6)	6088 (4)	4904 (3)	0·041
N(2)	6583 (6)	5515 (4)	6704 (3)	0·050
C(1)	2365 (8)	5337 (7)	5593 (4)	0·068
C(2)	2802 (8)	6190 (6)	5137 (4)	0·053
C(3)	4807 (8)	7044 (6)	4573 (4)	0·066
C(4)	4408 (9)	5298 (5)	4363 (3)	0·061
C(5)	6059 (8)	7309 (6)	6833 (4)	0·071
C(6)	6072 (8)	6318 (6)	7194 (4)	0·066
C(7)	8198 (8)	5544 (6)	6644 (4)	0·071
C(8)	6176 (9)	4515 (6)	6993 (4)	0·083
C(9)	7614 (9)	5020 (7)	5057 (4)	0·074
C(10)	8786 (7)	4442 (6)	4666 (4)	0·045
C(11)	9062 (8)	3436 (7)	4808 (4)	0·064
C(12)	10134 (12)	2938 (8)	4455 (6)	0·104
C(13)	10903 (12)	3403 (12)	3967 (6)	0·108
C(14)	10645 (13)	4397 (11)	3795 (4)	0·102
C(15)	9566 (10)	4928 (6)	4161 (5)	0·068
C(16)	1991 (7)	6975 (6)	7137 (3)	0·050
C(17)	758 (8)	7348 (5)	7587 (3)	0·042
C(18)	136 (10)	8298 (7)	7468 (5)	0·054
C(19)	−1006 (10)	8626 (6)	7867 (5)	0·070
C(20)	−1526 (8)	8033 (6)	8404 (5)	0·069
C(21)	−890 (8)	7115 (5)	8532 (4)	0·070
C(22)	235 (7)	6772 (5)	8127 (4)	0·056

Table 2. *Interatomic distances* (Å) *and angles* (°)

Cu–O(1)	2·179 (6)	O(4)–C(9)	1·212 (11)
Cu–O(2)	2·168 (6)	C(9)–C(10)	1·521 (11)
Cu–O(3)	2·126 (6)	C(10)–C(11)	1·388 (12)
Cu–O(4)	2·370 (6)	C(11)–C(12)	1·366 (14)
Cu–N(1)	2·060 (6)	C(12)–C(13)	1·328 (17)
Cu–N(2)	2·058 (6)	C(13)–C(14)	1·383 (21)
O(1)–C(1)	1·430 (9)	C(14)–C(15)	1·405 (15)
C(1)–C(2)	1·490 (12)	C(15)–C(10)	1·370 (12)
C(2)–N(1)	1·471 (9)	O(5)–C(16)	1·262 (8)
N(1)–C(3)	1·490 (10)	O(6)–C(16)	1·269 (9)
N(1)–C(4)	1·482 (8)	C(16)–C(17)	1·508 (9)
O(2)–C(5)	1·436 (10)	C(17)–C(18)	1·404 (12)
C(5)–C(6)	1·490 (11)	C(18)–C(19)	1·370 (13)
C(6)–N(2)	1·500 (10)	C(19)–C(20)	1·386 (13)
N(2)–C(7)	1·485 (9)	C(20)–C(21)	1·375 (11)
N(2)–C(8)	1·489 (10)	C(21)–C(22)	1·371 (10)
O(3)–C(9)	1·238 (10)	C(22)–C(17)	1·378 (10)
O(1)–Cu–O(2)	92·8 (2)	C(6)–N(2)–C(8)	108·8 (6)
O(1)–Cu–O(3)	106·3 (2)	C(7)–N(2)–C(8)	107·6 (6)
O(1)–Cu–N(1)	81·3 (2)	O(3)–C(9)–O(4)	123·6 (8)
O(1)–Cu–N(2)	95·1 (2)	O(3)–C(9)–C(10)	116·3 (8)
O(1)–Cu–O(4)	158·8 (2)	O(4)–C(9)–C(10)	120·1 (7)
O(2)–Cu–O(4)	105·7 (2)	C(9)–C(10)–C(11)	121·2 (7)
O(2)–Cu–N(1)	92·9 (2)	C(9)–C(10)–C(15)	118·9 (7)
O(2)–Cu–N(2)	82·5 (2)	C(10)–C(11)–C(12)	120·0 (8)
O(2)–Cu–O(3)	159·5 (2)	C(11)–C(12)–C(13)	120·6 (11)
O(3)–Cu–O(4)	57·1 (2)	C(12)–C(13)–C(14)	121·6 (11)
O(3)–Cu–N(1)	97·0 (2)	C(13)–C(14)–C(15)	118·7 (9)
O(3)–Cu–N(2)	88·6 (2)	C(14)–C(15)–C(10)	119·2 (8)
O(4)–Cu–N(1)	87·6 (2)	C(15)–C(10)–C(11)	120·0 (7)
O(4)–Cu–N(2)	97·4 (2)	O(5)–C(16)–O(6)	123·8 (6)
N(1)–Cu–N(2)	174·0 (2)	O(5)–C(16)–C(17)	119·2 (6)
		O(6)–C(16)–C(17)	116·9 (6)
O(1)–C(1)–C(2)	110·0 (6)	C(16)–C(17)–C(18)	120·4 (6)
C(1)–C(2)–N(1)	111·4 (6)	C(16)–C(17)–C(22)	120·8 (6)
C(2)–N(1)–C(3)	109·5 (5)	C(17)–C(18)–C(19)	120·3 (8)
C(2)–N(1)–C(4)	109·3 (6)	C(18)–C(19)–C(20)	120·1 (8)
C(3)–N(1)–C(4)	106·7 (5)	C(19)–C(20)–C(21)	119·6 (8)
O(2)–C(5)–C(6)	108·9 (6)	C(20)–C(21)–C(22)	120·8 (7)
C(5)–C(6)–N(2)	109·7 (6)	C(21)–C(22)–C(17)	120·4 (6)
C(6)–N(2)–C(7)	110·0 (5)	C(22)–C(17)–C(18)	118·8 (7)

and angles in Table 2.* The structure consists of $[Cu(C_7H_5O_2)(C_4H_{11}NO)_2]^+$ cations and benzoate anions. The O atoms of the uncoordinated benzoate ion are hydrogen bonded to the O atoms of the 2-dimethylaminoalcohols (Figs. 1, 2) with dimensions: O(1)–H(1) 0·90 (6), O(2)–H(2) 0·90 (8), O(1)···O(6) 2·569 (7), O(2)···O(5) 2·579 (7) Å, O(1)–H(1)···O(6) 171 (6) and O(2)–H(2)···O(5) 175 (8)°. Otherwise the ions are held in the crystal by van der Waals forces, all the other nonhydrogen intermolecular contacts being greater than 3·4 Å.

Each 2-dimethylaminoethanol ligand acts as a bidentate chelate through the N and O atoms, and the benzoate ion chelates asymmetrically through both O atoms. As far as is known this structure is the first metal complex in which 2-dialkylaminoethanol is coordinated as a neutral ligand. In the other complex

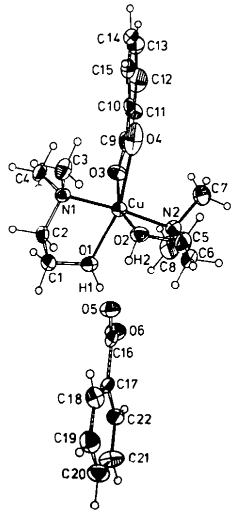

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the structure.

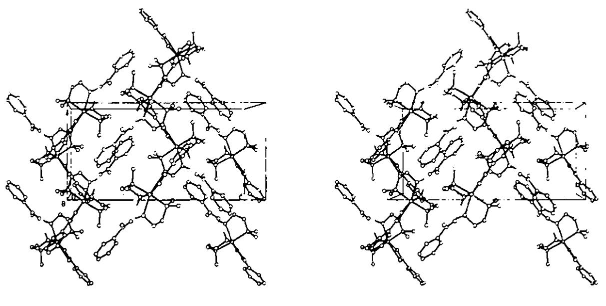

Fig. 2. Stereoview of the unit cell.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes' details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42444 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

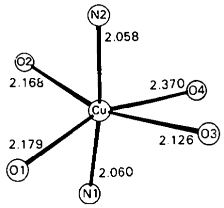

Fig. 3. The environment of the Cu atom viewed perpendicular to the plane defined by N(1),O(2),O(4).

structures, of which about thirty have been determined, the ligand acts as an anionic 2-dialkylaminoethanolato chelate.

The Cu atom is six coordinated, and the structure of the CuN_2O_4 core is shown in Fig. 3. The environment of the Cu atom is not typical for six-coordinate Cu^{II} chromophores (Hathaway, 1984) and represents a further example of the stereochemical variety found in Cu^{II} structures. On the basis of bond lengths and angles the environment of the Cu atom is perhaps best considered as a compressed octahedral stereochemistry. It should be added, however, that this classification is not unambiguous, because axial equatorial bond directions could be selected in a different way.

Except for the O(4)—C(9) bond of 1·212 (11) Å, the distances and angles within coordinated and uncoordinated benzoate groups are very similar and do not show a greater variation than would be expected on the basis of standard deviations. Distance and angular parameters of the 2-dimethylaminoethanol ligand are about as expected and do not warrant further comment.

References

Ahlgrén, M., Turpeinen, U. & Hämäläinen, R. (1982). *Acta Cryst.* **B38**, 429–433.

Hathaway, B. J. (1984). *Struct. Bonding (Berlin)*, **57**, 55–118.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980) *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Stewart, J. M. (1976). Editor. The *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Turpeinen, U., Hämäläinen, R. & Ahlgrén, M. (1980). *Acta Cryst.* **B36**, 927–930.

Acta Cryst. (1985). C**41**, 1730–1733

(4,7,13,16,21,24-Hexaoxa-1,10-diazabicyclo[8.8.8]hexacosane)sodium Tetratelluride(2—)

By Lesley A. Devereux and Gary J. Schrobilgen*

Department of Chemistry, McMaster University, 1280 *Main Street West, Hamilton, Ontario L*8*S* 4*M*1, *Canada*

and Jeffery F. Sawyer*

Department of Chemistry, University of Toronto, 80 *St George Street, Toronto, Ontario M*5*S* 1*A*1, *Canada*

(*Received* 4 *June* 1985; *accepted* 22 *July* 1985)

Abstract. $[Na(C_{18}H_{36}N_2O_6)]_2[Te_4]$, $M_r = 1309{\cdot}3$, monoclinic, $P2_1/c$, $a = 10{\cdot}710\,(5)$, $b = 12{\cdot}038\,(9)$, $c = 39{\cdot}14\,(2)$ Å, $\beta = 95{\cdot}07\,(4)^\circ$, $U = 5027$ Å^3, $D_x = 1{\cdot}73$ Mg m^{-3}, $Z = 4$, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}52$ mm^{-1}, $T = 295$ K, $F(000) = 2568$, $R_1 = 0{\cdot}082$ for 2660 observed $[I > 3\sigma(I)]$ reflections. Lattice contains cryptated Na^+ ions and tetratelluride anions, Te_4^{2-}, with terminal bond lengths 2·702 (3) and 2·702 (3) Å, a longer central bond of length 2·746 (3) Å, Te—Te—Te bond angles 109·7 (1) and 110·0 (1)° and a Te—Te—Te—Te dihedral angle of 105·3 (1)°.

Introduction. Elements of Group VI form anionic chains having the general formula Ch_x^{2-}. A large number of these anions have been structurally characterized, with $x = 3$–7 for S, 3–6 for Se, and 3–5 for Te (Huffman & Haushalter, 1984, and references therein). It is only recently that the structure of the third Te anion, Te_4^{2-}, has been determined in $[Ph_4P]_2[Te_4].2CH_3OH$ (Huffman & Haushalter, 1984). Each end of the tetratelluride(2—) anion is hydrogen bonded to a CH_3OH molecule. The present structure determination of Te_4^{2-} in $[(2,2,2\text{-crypt})Na]_2[Te_4]$ represents a Te_4^{2-} anion in an unsolvated environment. Significant differences in the gross structural features of the anions

---

* Authors to whom correspondence should be addressed.

0108-2701/85/121730-04$01.50